National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material 917a

D-Glucose (Dextrose)

Standard Reference Material (SRM) 917a is certified as a chemical of known purity. It is intended primarily for use in the calibration and standardization of procedures for glucose determinations employed in clinical analysis and for routine critical evaluation of the daily working standards used in these procedures.

Certified Concentrations in percent by weight:

Purity[a]	99.7 ± 0.2
α-D-Glucopyranose[b]	96.7 ± 0.2
ß-D-Glucopyranose[b]	3.0 ± 0.2

[a]The purity of SRM 917a, as total D-glucose, is based on allowance for the measured moisture and total oligosaccharide contents, and on the absence of impurities detectable by other methods. The uncertainty for purity is based on scientific judgment and is meant to approximate two standard deviations of the certified value.

[b]The uncertainties for the two glucopyranose compounds are two standard deviations of the certified values, and include a contribution for any observed sample variability.

Notice and Warnings to Users:

Expiration of Certification: This certification will be valid for five years from the date of shipment: Periodic reanalysis of representative samples from this SRM lot will be performed, and if significant changes are observed within the five year period, the purchaser will be notified by the National Institute of Standards and Technology (NIST). Please return the attached registration card to facilitate notification.

Storage: SRM 917a should be stored in the tightly closed, original container at 25 °C or lower. Refrigerated storage in a desiccator is recommended for prolonged storage. However, the bottle and contents should be allowed to warm to room temperature before opening. The material should not be subjected to heat or direct sunlight during storage. Under proper storage, experience at NIST indicates this material to be stable for at least five years.

Use: Samples should be weighed immediately after withdrawal from the bottle, which should be resealed tightly without delay.

The surface of the material absorbs a significant amount of moisture when exposed to a relative humidity of approximately 75 percent. Because the certified purity is based on a moisture content of 0.17%, any added moisture will lower the purity. Our experience indicates that moisture gain is not a significant problem at a relative humidity of approximately 50 percent.

SRM 917a IS INTENDED FOR "IN VITRO" DIAGNOSTIC USE ONLY!

Gaithersburg, MD 20899
August 31, 1989

Stanley D. Rasberry, Chief
Office of Standard Reference Materials

(over)

Noncertified values:

The following values are not certified and are provided for information only.

Moisture	0.17 ± 0.02	percent by weight
Total Oligosaccharide	0.16 ± 0.02	percent by weight
Ash	< 0.002	percent by weight

Specific Optical Rotation

$[\alpha]_D^{20}$ = 929.2 ± 0.3 mrad (53.24° ± 0.02°, at equil., ℓ 20.0 in water)

$[\alpha]_{546}^{20}$ = 1098.0 ± 0.3 mrad (62.91° ± 0.02°, at equil., ℓ 20.0 in water).

$[\alpha]_D^{20}$ = 1943.2 ± 1.4 mrad (111.34° ± 0.08°, initial, ℓ 10.0 in dimethylsulfoxide).

In these expressions, ℓ refers to the concentration in grams per 100 mL of solution.

The specific optical rotation measurements of the aqueous solutions were made without spiking with ammonia, using an automatic polarimeter that had been calibrated against internationally standardized quartz control plates, and also verified by reference to results from a high-precision polarimeter.

The uncertainties of the noncertified values are two standard deviations of the mean values, and include a contribution for any observed sample variability except for the total oligosaccharide. The listed uncertainty for the total oligosaccharide content is based on scientific judgment and is meant to approximate two standard deviations of the mean value.

Analysis: The contents of α-D-glucopyranose and ß-D-glucopyranose were determined from freshly prepared solutions of D-glucose in dimethylsulfoxide-d6 by ^{1}H NMR spectroscopy at 400 MHz and by ^{13}C NMR spectroscopy at 100.6 MHz. These methods revealed up to four isomers of D-glucose at various levels, two of which (furanose anomers), were formed on dissolution. No non-glucose impurities were detected. Moisture was determined by the Karl Fischer method. No impurities were revealed by thin-layer chromatography.

The total oligosaccharide content of the material was measured by high performance liquid chromatography. The possible presence of another impurity was indicated by a very weak ultra-violet absorption maximum at approximately 280 nm. However, the intensity of this absorption was substantially less than that of SRM 917.

Source: The D-glucose used for this SRM was obtained from Pfanstiehl Laboratories, Inc., of Waukegan, Illinois.

The analytical measurements leading to certification were performed in the NIST Center for Analytical Chemistry, Organic Analytical Research Division, by A. Cohen, B. Coxon, D.K. Hancock, S.A. Margolis, and L.T. Sniegoski.

The statistical analysis of the data was made by R. Paule, NIST National Measurement Laboratory.

The overall direction and coordination of the technical measurements leading to the certification were under the chairmanship of B. Coxon.

The technical and support aspects concerning the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by R. Alvarez.

Analytical Methods: For the certification of this SRM, the method used was isotope dilution/gas chromatography/mass spectrometry and involves converting glucose into a dibutylboronate acetate derivative. The method is considered to be "definitive" [1] for serum glucose by the National Committee for Clinical Laboratory Standards (NCCLS) [2].

Homogeneity Analysis: The homogeneity assessment was made at the time the certification analyses were performed. A stratified sampling plan was devised to test for homogeneity across the manufacturing process. The results indicated that there was no apparent trend in the data when plotted against the sequence in which the ampules were prepared.

REFERENCES

[1] "Development of Definitive Methods for the National Reference System for the Clinical Laboratory, Approved Guideline," NCCLS Publication NRSCL 1-A, National Committee for Clinical Laboratory Standards, Wayne, PA, (1991).

[2] White V, E., Welch, M.J., Sun, T., Sniegoski, L.T., Schaffer, R., Hertz, H.S., and Cohen, A., The Accurate Determination of Serum Glucose by Isotope Dilution Mass Spectrometry - Two Methods, Biomed. Mass Spectrom., **9**, pp. 395-405, (1982).

[3] *Guide to the Expression of Uncertainty in Measurement*, ISBN 92-67-10188-9, 1st Ed., ISO, Geneva, Switzerland, (1993): see also Taylor, B.N. and Kuyatt, C.E., "Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results," NIST Technical Note 1297, U.S. Government Printing Office, Washington, D.C., (1994).

[4] U.S. Department of Health and Human Services, "Biosafety in Microbiological and Biomedical Laboratories," U.S. Government Printing Office, Washington, D.C., (1988).